Microwave Facilities for Welding Thermoplastic Composites and Preliminary Results

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Abstract: The wide range of applications of microwave technology in manufacturing industries has been well documented (NRC, 1994; Thuery, 1992). In this paper, a new way of joining fibre reinforced thermoplastic composites with or without primers is presented. The microwave facility used is also discussed. The effect of power input and cycle time on the heat affected zone (HAZ) is detailed together with the underlying principles of test piece material interactions with the electromagnetic field. The process of autogenous joining of 33% by weight of random glass fibre reinforced Nylon 66, polystyrene (PS) and low density polyethylene (LDPE) as well as 23.3 % by weight of carbon fibre reinforced PS thermoplastic composites is discussed together with developments using filler materials, or primers in the heterogenous joining mode. The weldability dependence on the dielectric loss tangent of these materials at elevated temperatures is also described.

1. Introduction

Industrial applications of microwaves are relatively new technology. Factors, like unfavourable socioeconomic conditions, a natural reluctance to accept an entirely new, unproven process, a lack of experience and so on, that hinder the use of microwaves in materials processing are declining so that prospects for the development of this technology seems to be very bright (Sutton, 1989). The general mechanisms that govern the energy dissipation process and the microwave/material interaction include dipole friction, current loss and ion jump relaxation (Metaxas and Meredith, 1983; Siores, 1994).

The growth in using thermoplastic composites as structural materials remains very strong (Schwartz, 1992) and welding technology development fuels that growth. The advantages of using thermoplastic composites over the frequently used thermosetting composites include their capability to be formed into complex shapes at lower costs and higher productivity rates

(Varadan and Varadan, 1991; Partridge, 1989). They are light, corrosion resistant, possess high dimensional accuracy for parts, greater damage tolerance and can be moulded and remoulded. Most thermoplastic composites are joined by fusion bonding and the processes employed include resistance welding, ultrasonic bonding, vibrational bonding, high frequency welding, traditional infrared heated air, hot plate, hot melt and room-cure adhesives. The advantages and limitations of these joining processes have been fully documented in the literature (Schwartz, 1992). The frequency range used in the high-frequency welding is 3 to 40 MHz which is in the radio frequency range (Schwartz, 1995). However, three papers (Varadan and Varadan, 1991; Strokes, 1989; Wu and Benatar, 1992) on microwave joining of thermoplastic composites have had a considerable impact as far as this emerging technology is concerned. The merits of employing microwaves in joining thermoplastic composites include having a clean and reliable interface at the joints, fast joining time with minimum destruction of the properties of the bulk materials and limitations encountered in other processes are avoided (Varadan and Varadan, 1991).

2. Microwave and Peripheral Facilities

The prototype equipment used for the study is shown in Figure 1. The microwaves generated from the magnetron were guided through a WR340 waveguide to the test pieces. The primary concern was to avoid radiation leakage, therefore the welding process was enclosed within a shielded cavity. This equipment was built around a modified commercial microwave oven. The 0.8 kW magnetron was relocated on top of the oven cavity via a piece of WR340 waveguide. Another piece of waveguide with slits opened for positioning the test pieces was placed upright within the oven cavity. The upper end was fitted with a flange

connected to the magnetron mounted on top of the oven. The lower end was similarly attached to an additional length of waveguide containing a shorting plunger.

With reference to Figure 1, the incident waves generated by the magnetron travelled downwards through three sections of WR340 waveguide and interacted with the test pieces located in the second section before being reflected back by the top face of the adjustable plunger. The plunger was designed and manufactured to have a sliding fit contact with the waveguide. The interaction between the incident and the reflected waves set up a standing wave (Glazier and Lamont, 1958) and as desired the maximum electric field occured at the seam of the butted or lapped test pieces. This was achieved by adjusting the moveable piston so that its top face was an odd multiple of $\lambda_g/4$ from the centre of the slit, λ_g being the wavelength within the waveguide.

2.1 Short-Circuit Plunger and Its Dimensions

The relationship between the wavelength within the guide, λ_g, and the free space wavelength, λ_o, was found to be related as follows (Glazier and Lamont, 1958):

$$1/\lambda_g^2 = 1/\lambda_o^2 - (1/2a)^2 \qquad (1)$$

where a is the larger of the internal dimension of the waveguide expressed in mm.

It was calculated that : $\lambda_o = (3\text{x}10^{11})/(2.45\text{x}10^{9}) = 122.45$ mm.

For the waveguide used, WR340 having a = 86.36 mm and b = 43.18 mm,

therefore, $\lambda_g = 173.63$ mm and $\lambda_g/4 = 43.408$ mm.

Referring to Figure 2, the distance between the centre of the slit and the top face of the plunger, *l*, was initially 286 mm. To establish a maximum electric field, *l* had to be varied

so that l = n x $\lambda_g/4$ with n being an odd integer. When n = 7 was chosen then l = 7 x $\lambda_g/4$ = 7 x 43.408 mm = 303.9 mm. The distance, l, could be varied by adjusting the plunger up and down by rotating the knob at the bottom of the plunger. Hence, in order to establish the maximum electric field at the butted specimens interface, the plunger was adjusted by 303.9 mm - 286 mm = 17.9 mm in the downward direction. This was the position of the electric field maximum where the test pieces being at room temperature and formed part of the initial set-up.

Leakage was expected because of the sliding fit, and measures were taken to prevent it. It was minimised by using a non-contact method to produce an apparent short circuit at the front face of the plunger. The actual point of contact was arranged to be at a point where effective current was zero. This was accomplished as shown in Figure 3. At a distance of $\lambda_g/4$ from the top face of the piston the traverse electric field, E had a maximum value and the traverse magnetic field H was zero (Glazier and Lamont, 1958). Thus the ratio of traverse components E/H was infinite at this point and hence the input wave impedance of a $\lambda_g/4$ length of waveguide closed at its far end was infinite. Similarly, the input impedance of a $\lambda_g/2$ length was zero. In practice, however, neither the infinite nor the zero values can be attained.

In Figure 3, the part ABC is regarded as a narrow waveguide of length $\lambda_g/2$ short-circuited at C and folded at its centre , B. Thus the input impedance at A became zero, and there was an equivalent short-circuit at the front face of the piston which therefore appeared as a continuous short-circuit across the waveguide. The sliding contact occured at B and at a distance $\lambda_g/4$ from the end, point C. At point B, the impedance was relatively high and thus both the magnetic field and the current were small (Glazier and Lamont, 1958). As a result,

radiation leakages through the sliding contact were extremely small. Variations in contact resistance with movement of the piston were of little importance. Here, the actual physical short-circuits were replaced by virtual short-circuits and physical contact was established at a place where poor contact was not critical.

An example (Rizzi, 1988) for the dimensions of the moveable plunger is shown in Figure 4, where, b = 20 mm, b_1 = 0.6 mm b_2 = 4.0 mm.

Therefore, in the WR340 waveguide configuration, b = 43.18 mm and by proportion,
b_1 = 0.6/20 x 43.18 mm = 1.30 mm and b_2 = 4.0/0.6 x 1.30 mm = 8.636 mm.
Since the plunger had a sliding contact with the waveguide, the clearance between these two components was made to be 0.5 mm. The angle of 45^o was chosen for convenience of manufacture. The dimensions of the plunger are shown in Figure 5.

3. Materials/Microwaves Interaction Considerations

This section presents a brief theoretical overview of the interaction of electromagnetic waves and hence microwaves with matter. Consideration is given to the transmission of microwaves in free space, along waveguides and within matter. Interactions at the fibre reinforced thermoplastic composite/air interface is also considered. Maxwells Equations (Kraus, 1992) are normally used to analyse the interaction phenomena between microwaves and materials.
An electromagnetic wave propagates through empty space at the velocity of light. In such a wave the time varying magnetic field may be regarded as generating a time changing electric field, which in turn generates a magnetic field and the process is repeated. Microwaves are electromagnetic waves that have a frequency range of 0.3 to 300 GHz with corresponding

wavelengths ranging from 1 m to 1 mm. With respect to the Cartesian coordinate system, x, y, z, a harmonic wave propagating down the z axis can be written as a scalar equation :

$$d^2E/dt^2 = c^2 (d^2E/ dz^2) \quad (2)$$

where : E is a vector in the xy plane; and

c is the velocity of electromagnetic wave in vacuum and is related to the electromagnetic properties of the medium by :

$$c^2 = 1/(\mu_0 \varepsilon_0) \quad (3)$$

where : μ_0 is the absolute permeability of free space ; and

ε_0 is the absolute permitivity of free space.

Random glass fibre reinforced (33%) Nylon 66 [Nylon 66/GF(33%)] was chosen for the study because the loss tangent at room temperature (Michaeli, 1995) of Nylon 66 is high compared with other commonly used thermoplastic materials. Random glass fibre reinforced (33%) LDPE [LDPE/GF (33%)] was also selected because there was a successful case (Wu and Benatar, 1992) of welding the composite with HDPE as a matrix using microwave energy and it was believed that LDPE would couple better to microwaves (NRC, 1992) as its cyrstallinity is lower than that of HPDE. Polystyrene (PS) matrix was chosen because first, it is a common thermoplastic polymer matrix (Shackelford, 1994) and second, its loss tangent (Michaeli, 1995) is very near to that of LDPE thus, a comparison could be made later on.

In all cases, the length of the reinforcing fibre was 6 mm or less as previous investigations showed that optimum mechanical properties (Lubin, 1982) could be attained when 6 mm fibres were used and there were only marginal differences in mechanical properties if fibre lengths between 3 mm and 6 mm were used. The test pieces were injection-moulded to shape.

The advantages and limitations of using adhesives as fillers are numerous (Everett, 1995; Waters, 1996; Black, 1996), but only a few are mentioned here. One of the major advantages of adhesive bonding is that the loads imposed on the joint are spread evenly over the whole joint area. Another important advantage is that almost all engineering materials can be joined to one another by adhesives.

One of the major shortcomings is the requirement for surface preparation. Surfaces have to be roughened to provide an improved adhesive key (Waters, 1996) and to be cleaned (Black, 1996; Bolger, 1983) for consistent high quality joint performance. Another shortcoming is the relatively low operating temperature for the final products; the maximum operating temperature for the adhesive used is only 160^{o}C (Selleys, undated) but some other adhesives may have operating temperatures of up to 300 ^{o}C (Waters, 1996). A butt joint was initially chosen for the welded connections between the two half test pieces. The bonding was partially successful because of 'thermal runaway', which gave rise to slippage between the butted interfaces. The partially bonded test pieces were discarded.

4. Test Piece Microwave Interaction Results

The two mirror image test pieces were cut using a band saw from a standard tensile test piece for composite materials. The interface was then roughened by rubbing it against a piece of coarse, grade 80, Emery paper. Butt joints were first selected for bringing two parts together for joining and all composite test pieces were joined together by 2 cc of adhesive. Ten grams of graphite powder were sprayed onto the side faces of the test pieces, up to 20 mm from the interface other than those of carbon fibre (CF) reinforced PS. The test piece was then located, spring loaded and clamped. The spring was made to push the two pieces

aligned together when the interface was melted by microwave energy. The spring force was kept to about 10 N. The composite test pieces were welded using 240 W constant power and 4 seconds of continuous exposure time. The HAZ for the CF reinforced PS was on the upper part of the test pieces contained in the waveguides ie 21.5 mm from both sides of the interface. When the exposure time was increased to 5 seconds, the spring forced the two pieces to bend to a vee- shape with the lower part of the joint being broken.

Test pieces of other materials were found to have 20 mm from both sides of the interface as their respective HAZs and this was totally due to the amount of graphite powder applied. When the exposure time was increased to 5 seconds, the graphite powder flashed resulting in a burnt zone formed at the interface between the test pieces. The graphite powder was over-heated and glowed as a result of 'thermal runaway' due to localised heating of the test pieces. Tensile tests were carried out with the successfully welded test pieces and it was found that the bond strengths of all materials ranged from 8.5 % to 24.3% of those of the respective parent materials. Results are shown in Figure 6.

A Shimadzu tensile testing machine was used for the test having a load range of 2 KN and a maximum load rate of 600 N per minute (Bolton, 1996). After being tensile-tested, the interfaces of the broken test pieces were investigated and the adhesive on the interface was ground away using a tool grinder. In 33% glass fibre reinforced PS [PS/GF (33%)] test pieces, black marks were observed on the grey interface and were estimated to occupy up to 45% of the interface area. The marks appeared to be similar to those found at the interface of the same material after the material was cut using a metallic bandsaw. After removing several thin layers of the interface, the marks still appeared and the measured thickness was found to be 2 mm. It was deduced that the marks were left behind after burning and melting

of the composite and that welding of the material did occur but was spread over only up to 45% of the interface area. In this case, it could be argued that the expected bond strength of the welded part could be up to 54% of the parent material.

However, it was difficult to locate similar black marks on the same position in the case of welded nylon 66/GF test pieces because the base material was black. Similarly black marks were also traced when LDPE/GF test pieces were joined and the corrected bond strength of the weld was found to be up to 48% of the parent material.

Another primer used was a five minute curing time two part adhesive containing 100% liquid epoxy and 8% amine, which is microwave reactive (NRC, 1994). The first three factors (Everett, 1995) to be considered in using adhesives as primers are their shelf life, port life and closed assembly time. The shelf life of the two-part adhesive used was infinite, provided that the two parts did not come into contact with one another; its port life was only five minutes. Its closed assembly time has not been fully investigated. It appeared that it was too long.

Simple lap joints were then selected for the connection of the two half test pieces and PS/GF(33%) was first selected for the study. The lapped area was made to be 20 mm x 10 mm. The maximum linear overlap of the test pieces was 20 mm with which the increase in overlap length brought about a linear increase in shear strength (Everett, 1995; Bolger, 1983). The lapped areas were first roughened by rubbing them against coarse, grade 80, Emery paper. They were then cleaned by immersing them in methanol 10 % solution and were allowed to dry in air before applying primer onto them. After applying the filler, the two pieces were tightly assembled . This was in order to fix the relative position between

the two test pieces and to apply sufficient pressure onto the lap joint so that the adhesive thickness remained constant for all specimens. The pressure on the lap joint was 10 N and it was critical as the bond strength of the test pieces cured by leaving them in ambient conditions for 16 hours (Selleys, undated) with and without assembly pressure were 611 N and 335 N respectively. In other words , the shear stresses were 3.055 N/mm^2 and 1.675 N/mm^2 respectively. The bond strength obtained for the GF reinforced thermoplastic/GF reinforced thermoplastic bond was low as compared with that produced by the best metal/metal bond (Everett, 1995). The former was only 21.8 % of the latter.

Subsequent tests were performed with the test pieces exposed to two different power levels of 400W and 800W with varying time intervals of microwave exposure. In all cases, only the parts smeared with filler were warmed or heated depending on the power level used and the time of microwave exposure. The test pieces were allowed to cool down to room temperature before being shear tested to obtain maximum bond strength measurements. The results are summarised in Figure 7. With reference to Figure 7, it was found that at 400 W, the peak bond strength was achieved by exposing the test pieces to microwave energy for 2 minutes; the bond strength (651N) and hence the shear stress (3.255N/mm^2) at this exposure duration exceeded that obtained by ambient conditions (conventional) curing by 17%. However, the time required was a mere of 0.2% of its counterpart. For exposure times of one and a half to four and a half minutes, the shear stresses obtained using microwave-cured filler were higher than those obtained by allowing the adhesive to set under ambient conditions. The maximum bond strength (661N) and hence the maximum shear stress (3.305 N/mm^2) were achieved at 800 W when the exposure time was 45 seconds and it exceeded the ambient conditions cured bond strength by 19 %; but the time required was only 0.08% of its counterpart. The lower bond strength obtained, for the test pieces exposed

to microwaves for over 2 minutes and 45 seconds for power levels of 400 W and 800 W respectively, may have been due to over-curing of the adhesive.

The PS/GF (33%) test piece was then selected and exposed to 800 W for 45 seconds. It was observed when viewed from the side of the test piece that outgassing had taken place and traces of white 'bubbles' resulting from the release of dissolved air at elevated temperature were found. The white bubbles contained the primer and its resulting colour implied that the filler had cured normally. Another test piece of PS/GF(33%) was exposed to 800 W for 75 seconds and it was found that the adhesive was charred, puffed up, leaving behind a yellowish resin mass; the yellowish colour suggested that the primer was over-cured. A similar phenomenon has been described in the literature (Bolger, 1983) with a one-component epoxy adhesive, dicyandiamide.

The HAZ seemed to be confined to the lapped area, beyond which the heating effect of microwave energy was not encountered. Shear tests revealed that 60% of the failures were due to the failure of the adhesive at the joint interface. The remaining failures took place in the original materials (GF/PS), which had an average strength of 1423 N or tensile strength of 47.43 N/mm^2. This meant that the adhesive bond strength exceeded the ultimate strength of the adherend and this case was common when thin test pieces are used (Bolger, 1983). On the other hand, test pieces thicknesses used in this research were not too thin, so it could be argued that microwave energy played some role in increasing the shear strength of the material. At 400 W of power and a 2 minute exposure time, the highest bond strength achieved was 720 N; whereas at 800 W and 45 second exposure, the peak bond strength obtained was 905 N. It was therefore argued that the higher the power level used, the shorter the exposure time required to achieve higher bond strength. These were up to 29 % and 62 % respectively stronger than the conventionally cured test pieces.

With Nylon 66/GF (33%), the peak bond strengths obtained at exposure times of 35 and 55 seconds for 400 W and 240 W respectively are depicted in Figure 8. They were 32% and 28% respectively higher than those obtained by curing the adhesive at room temperature conditions but the times required were only 0.06% and 0.1% of their counterparts. This material together with the adhesive seemed to couple better with microwaves than PS/GF (33%). With reference to Figure 9, microwave exposure time of over 42 seconds at 400 W burned the test pieces. At 240 W, burning of test pieces occurred at an exposure time of over 62 seconds. When exposed to 65 seconds, the test pieces burned mildly and diffusion of parent material into the filler became more prominent. This was examined using low power microscopy; three photos of the examination were taken and shown in Figures 10, 11 and 12. The bond strength was also higher than that exposed to 62 seconds. The bond strengths at an exposure time of 70 seconds were even higher than those exposed to 65 seconds. However, the test pieces were more seriously burnt and this weakened the parent material.

Referring to Figure 9, at 800 W, the cluster of bond strengths at microwave exposure times ranging from 25 to 40 seconds were best represented by the line 800PE1, ie. their average value; while those resulting from microwave energy exposure in the range of 45 to 65 seconds were averaged in line 800PE2. In both cases, results obtained were similar to the work using high density polyethylene (HDPE) (Siores and Groombridge, 1997). A step function was formed; at shorter exposure times, the average bond strength was only 97% of that cured under ambient conditions and it could be argued that no diffusion of parent material to the primer had taken place. When longer exposure times were used, the average bond strength was 41% higher. The processing times were also merely 0.06% and 0.1% respectively of the conventional ones. At 400 W, the cluster of bond strengths obtained by exposing to microwaves from 135 to 240

seconds were best represented by the line 400PE1. Their average value was 18% higher than that cured under ambient conditions and the processing time was only 0.33% of its counterpart. The average bond strength of test pieces cured conventionally was relatively low, 311 N, as stated by the manufacturer's instructions (Selleys, undated) for the adhesive.

Figure 13 shows the bond strengths of the three types of materials procured by exposing the test pieces at different intervals at 400 W. It was found that the peak or average bond strengths of Nylon 66/GF(33%), LDPE/ GF(33%) and PS/GF(33%) were 32%, 18% and 17% respectively higher than those cured in ambient conditions. The effects of the 33% glass fibre reinforcement and the primer on the loss tangent, tan δ, and the complex permittivity, ε, of the materials were assumed to be the same for the three types of thermoplastic composite materials. The two dielectric properties of the materials could therefore be used to explain the characteristics of the bond strengths of the composites as depicted in Figure 13.

The loss tangents (Metaxas and Meredith, 1983) of nylon and polyethylene were 0.0119 and 0.00116 respectively, subject to variations with the presence of impurities and their dielectric constants (Metaxas and Meredith, 1983) were 3.02 and 2.25 respectively. The loss tangent (Michaeli, 1995) of polyethylene was also higher than that of polystyrene. Nylon with its higher loss tangent and dielectric constant was best coupled with microwaves and this was illustrated by the short joining times as well as the highest increase in bond strength. By plotting the bond strengths of the two types of materials, obtained by exposing test samples at 800 W for different microwave exposure times, as shown in Figure 14, it was found that the results matched the loss tangent values of the two thermoplastic composite materials. The adhesive had been found by other researchers to be unsuitable for gluing polyethylene under ambient conditions (Selleys, undated). The relatively low bond strength obtained in ambient-condition cured primer with

LDPE made that procured by microwave processing look promising. The application of microwave energy in this particular case was therefore very successful because it caused significant improvement in bond strength as a result of the diffusion (refer to Figures 10, 11 and 12) of the parent material to the filler as described in more detail in the following section.

4. Bond Surface Analysis

The bond strengths encountered at some exposure time were higher than those cured conventionally because the primer diffused into parent material and vice versa at the interface. This phenomenon was further confirmed by the x-ray photoelectron spectroscopy surface analysis. Figure 15 shows the O and C atom intensities against binding energy of LDPE/GF (33%) plus araldite after the material has been exposed to 800 W microwave energy for 70 seconds. Figure 16 shows the –C-C- and -C-O bond intensities against binding energy of LDPE/GF (33%) plus araldite. From the two figures, the percentage of different atoms present in LDPE/GF (33%) plus araldite was worked out and shown in Table 1. Figure 17 illustrates the O and C atom intensities against binding energy of araldite on glass. Figure 18 depicts the –C-C-, -C-O and –C=O bond intensities against binding energy of araldite. From the two figures, the percentage of different atoms in araldite was worked out and shown in Table 2. The C atom intensity against binding energy of LDPE was shown in Figure 19 and no oxygen atom was found. Figure 20 illustrates the –C-C- bond intensity against binding energy of LDPE. The percentage of atoms of the three different materials were studied and summarised in Table 3.

Composite 1was formed by having some araldite mixed into LDPE/GF(33%).

Composite 2 was formed by having some LDPE/GF(33%) melted into araldite.

In fact Composite 1 is the same as Composite 2 and only differs by the name in which they are expressed. The relative percentage of oxygen in Composite 2 (7.9 %) was lower than that in araldite (13.8%). This was due to melting of LDPE/GF(33%) and subsequently transferring into the araldite. The relative amount of oxygen was reduced. The relative percentage of oxygen in Composite 1 (7.9%) was higher than that in pure LDPE (0%) because some araldite had entered this composite. It can be argued that oxygen came from the reinforcing glass fibre and in this case the oxygen content of Composite 1 should only be four times the percentage of silicon, ie. 2.4 percent. However, this was not the case and the oxygen was also present in 'C-O' form which indeed was present in araldite. So it can be deduced that the oxygen content in Composite 1 is mainly due to the araldite. LDPE/GF(33%) was transferred into the araldite.

6. Conclusions.

From the results obtained, it can be argued that the industrial application of microwave technology to the joining of thermoplasic composite materials shows promise. The potential benefits of the technology include speeding up the replacement of thermosetting resins by advanced thermoplastic composites in the structural parts of aeronautical, military and recreational industries.

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